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This paper presents the results of one phase of research carried out at the Jet Propulsion Laboratory, California Institute of Technology, under Contract No. NAS 7-100, sponsored by the National Aeronautics and Space Administration.

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TENSILE PROPERTIES OF PYROLYTIC TUNGSTEN FROM 1370° TO 2980°C IN VACUUM*

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SUMMARY

The tensile properties of annealed pyrolytic tungsten were measured at a strain rate of 0.02/min from 1370° to 2980°C in vacuum. Data for powder metallurgy and plasma-flame single-crystal tungsten are shown for comparison. A few engineering stress–strain curves are given for pyrolytic and powder metallurgy tungsten. Although voids are present in the pyrolytic material after annealing, the voids which form and grow during testing have a more important detrimental effect on ductility. The "as deposited" anisotropy of the microstructure is maintained after recrystallization and enhances the tendency to void formation and growth. With the exception of one specimen which contained no voids, as the result of a lower annealing temperature, pyrolytic tungsten has a lower ultimate strength than powder metallurgy tungsten in the temperature range studied.

INTRODUCTION

Pyrolytic tungsten, or tungsten deposited from a vapor phase on a heated substrate, is a newer form of tungsten that presents manifold possibilities for shaping because it may be deposited on complex mandrels. It is of interest to know how the pyrolytic material compares with tungsten in other forms. Some tensile properties of pyrolytic tungsten from 1370° to 2980°C are presented and a comparison is made with powder metallurgy (PM) tungsten and plasma-flame single-crystal (PF) tungsten.

MATERIALS

Quarter-inch plates of pyrolytic tungsten***, which became available in the past year, were used to make the R-4 type tensile specimens[1] used in this laboratory. Deposition rates from tungsten hexafluoride carried in hydrogen gas were reported by the producer to be from 0.005 to 0.010 in./h on a graphite substrate held at 650°C. Figure 1 shows how a specimen was cut from a plate and the orientation of the grains with

* This paper presents the results of one phase of research carried out at the Jet Propulsion Laboratory, California Institute of Technology, under Contract No. NAS 7-100, sponsored by the National Aeronautics and Space Administration.
** Formerly 1st Lt. U.S. Army, detailed to Jet Propulsion Laboratory. Present address: Pratt and Whitney Aircraft Co., North Haven, Connecticut.
*** Material furnished by High Temperature Materials, Inc., Boston, Mass., U.S.A.

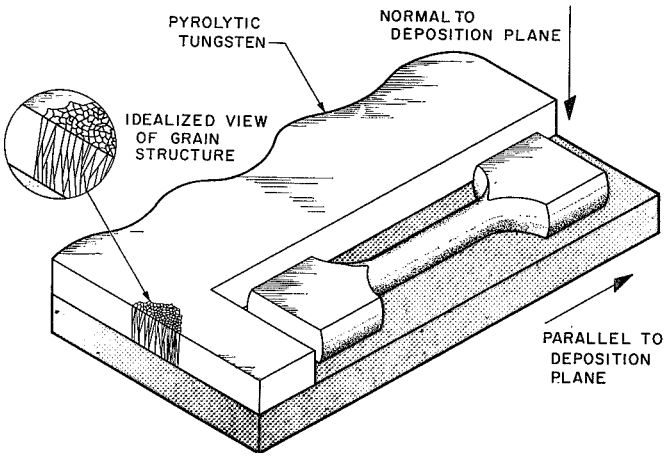


Fig. 1. Specimen orientation in pyrolytic tungsten.

respect to the tensile axis. Sufficient material was removed to eliminate any possible contamination from the substrate. This specimen, with modified head, had a 0.160 in. diameter and 0.640 in. gauge length. Photomicrographs (Fig. 2) of pyrolytic tungsten show a microstructure characteristic of vapor-deposited material.

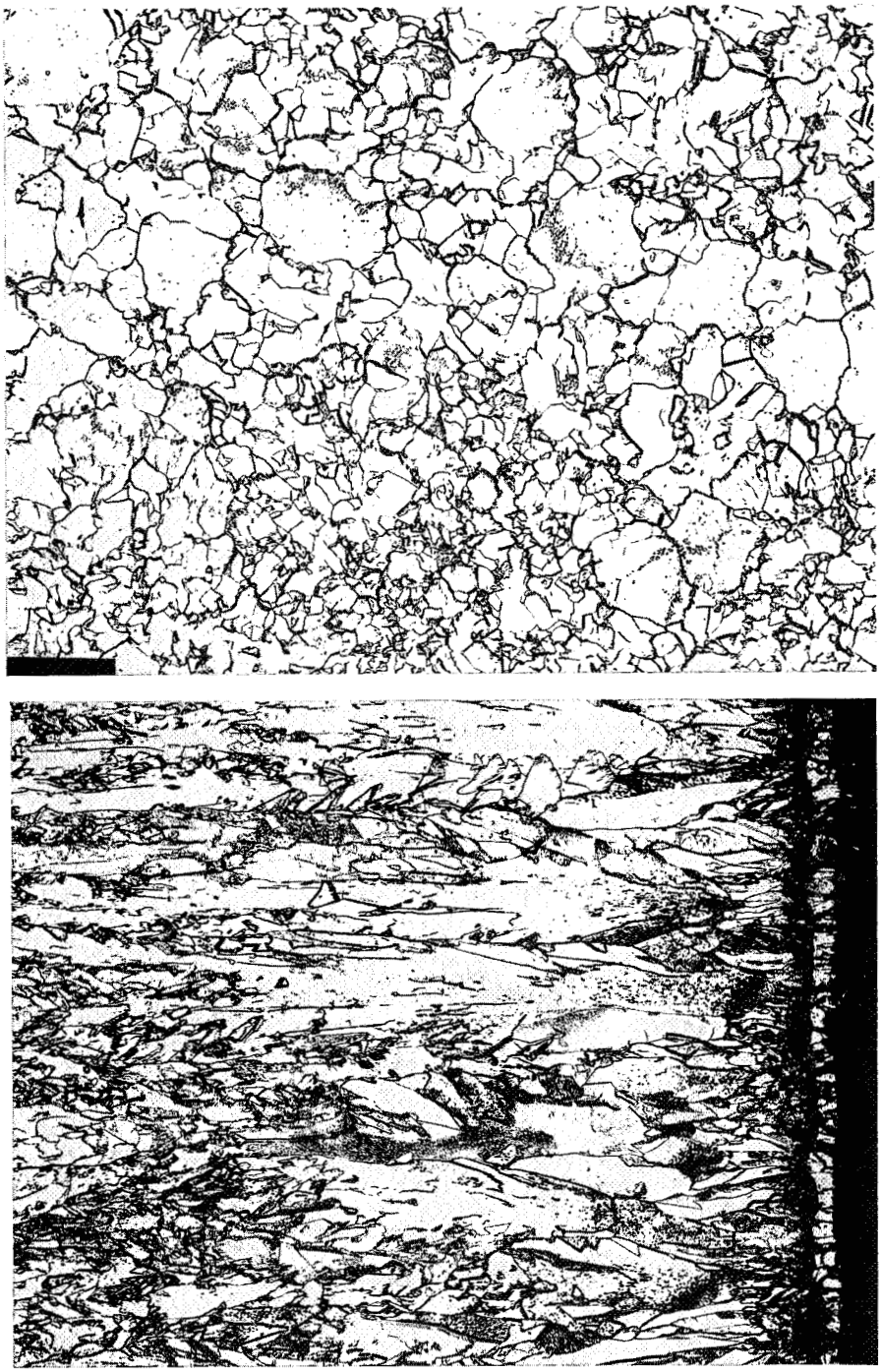
Comparison of the composition of pyrolytic tungsten with PM and PF tungsten is made in Table I. Pyrolytic tungsten has approximately equivalent contents of oxygen and nitrogen and a carbon content intermediate between the PM and PF materials, all in the "as received" condition. The contents of substitutional impurity elements in pyrolytic tungsten are lower, presumably because of the high purity of the tungsten hexafluoride from which it has been produced.

TABLE I

CHEMICAL COMPOSITION OF TUNGSTEN MATERIALS

Element	*Concentration (p.p.m.)*					
	Pyrolytic				*Powder metallurgy (PM)*	*Plasma flame (PF)*
	*As received**	*2000°C anneal*	*As received*	*2845°C anneal*	*As received*	*As received*
Oxygen	12	12	4.9	3.2	1.3–5	5–9
Carbon	17	17	31.0	15.4	2–4	66–98
Nitrogen	10	10	4.0	4.0	5–16	3–7
Hydrogen	2	2	1.1	1.8	---***	---
Fluorine	10	10	74	70	---	---
Silicon	10	1	<10	<10	10–20	20–40
Iron	10	10	<10	<10	10–40	---
Molybdenum	ND**	ND	<10	<10	30–50	---
Aluminum	10	1	<10	<10	20–40	10–30

* Representative analyses supplied by High Temperature Materials, Inc. (Other analyses from a commercial laboratory.)
** ND = Not detected.
*** --- = Not determined.



(a)

(b)

Fig. 2. Pyrolytic tungsten "as deposited". Etched 1 min in 30 cm^3 lactic acid, 10 cm^3 HNO_3, and 10 cm^3 HF (× 100). (a) Section parallel to deposition plane. (b) Section normal to deposition plane showing graphite substrate.

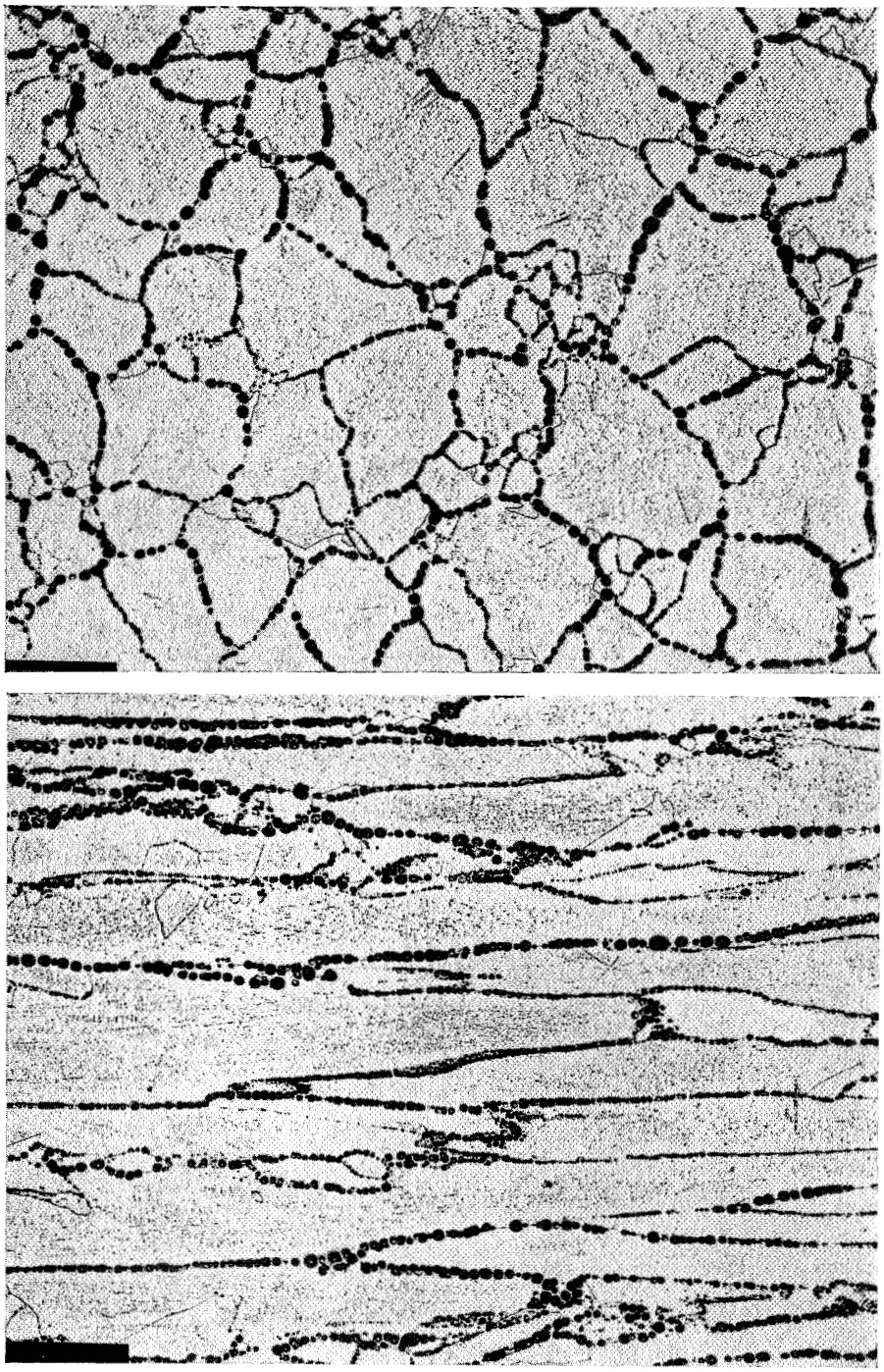


Fig. 3. Pyrolytic tungsten annealed 10 min at 2845°C. Etched 1 min in 30 cm^3 lactic acid, 10 cm^3 HNO_3, and 10 cm^3 HF (× 100). (a) Section parallel to deposition plane. (b) Section normal to deposition plane.

APPARATUS AND PROCEDURE

Testing apparatus and procedure have already been described[2]. Principal features include an induction-heated tungsten susceptor with a hot-grip assembly for holding the specimen on shoulder fillets, and load–elongation recording instrumentation. Vacuum employed in this study was of the order of 10^{-4} torr. All testing was carried out at a strain rate of 0.02/min.

Prior to testing, all specimens were heated in vacuum to 2845°C for 10 min to stabilize the structure for testing up to this temperature. The PM and PF tungsten materials used for comparison were similarly heat treated[3]. In the pyrolytic material, the 10 min treatment resulted in grain growth and the formation of voids (Fig. 3). The anisotropy of the deposit was maintained during grain growth. The volume of voids in the "as heat-treated" structure (Fig. 3) is between 5 and 10% as estimated by the line intercept method. The voids may result from fluorine entrapped during deposition.

RESULTS AND DISCUSSION

Typical stress–strain curves are shown in Fig. 4 for the pyrolytic and PM material. Pyrolytic tungsten has lower strength and lower elongation at all test temperatures, although the curves show similar shapes at higher temperatures. Above 1930°C, the relatively long region of decreasing stress prior to fracture in both materials is of interest. Although this decrease could possibly be explained by void growth during testing, similar decreasing stress regions were found in the curves for PF tungsten and also for PM tungsten tested at higher strain rates[3]. Neither exhibited void formation or growth.

A summary of the tensile data is given in Figs. 5–7. Figure 5 shows the ultimate tensile strength of pyrolytic tungsten as a function of temperature. The pyrolytic tungsten has lower strength than the PM material at all temperatures and higher strength than the PF material up to 2750°C. The initial void volume in the "as heat-

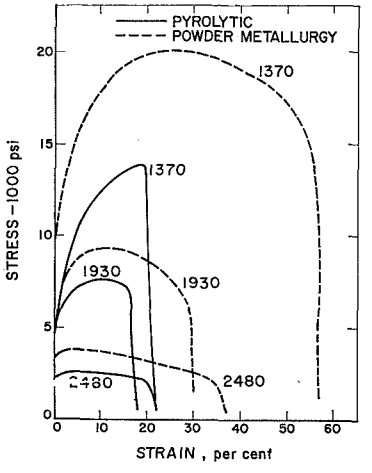


Fig. 4. Typical stress–strain curves at indicated temperatures (°C) for pyrolytic and powder metallurgy tungsten.

treated" pyrolytic tungsten could account for the strength difference relative to PM tungsten. The pyrolytic tungsten, even with voids, is stronger than the PF tungsten. The lack of grain boundaries in the PF material probably accounts for its lower strength. The relative effect of compositional differences is unknown.

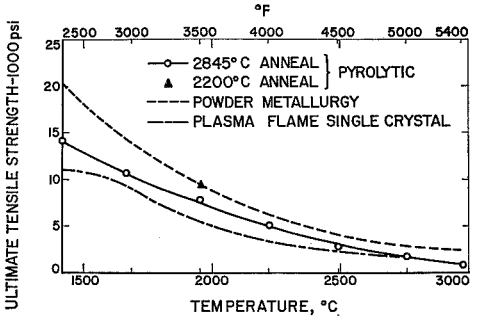


Fig. 5. Ultimate tensile strength of pyrolytic tungsten.

The lower reduction-in-area values for the pyrolytic and PM tungsten as compared to the PF tungsten (Fig. 6) are attributed to the effect of internal voids associated with intercrystalline fracture. In the pyrolytic material, this results from the voids present after annealing in combination with void growth during testing. Metallographic studies revealed an increase in void volume with testing. Fracture occurred in the pyrolytic tungsten in an intercrystalline manner at all test temperatures with

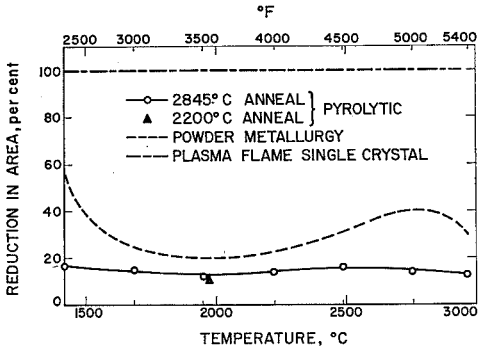


Fig. 6. Reduction in area of pyrolytic tungsten.

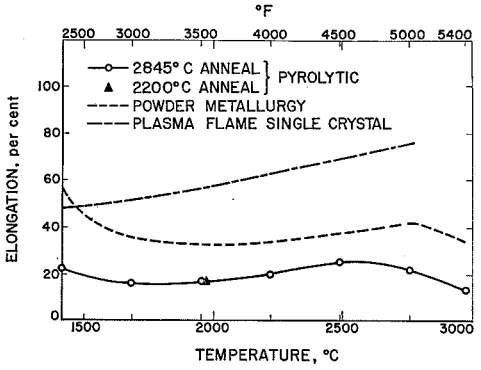


Fig. 7. Elongation of pyrolytic tungsten.

little or no necking. As shown in Fig. 8, the microstructure of a pyrolytic specimen tested at 1930°C contains a void volume of approximately 18% in a section away from the fracture. Void growth and intercrystalline fracture were found in the PM material at 1650°C and above[3]. No voids are present in PF material. The predominance of grain boundaries perpendicular to the tensile axis in the pyrolytic material would be expected to increase the tendency toward void formation during testing when compared with equiaxed grains of the same grain size in PM material.

Although the volume of voids formed during testing increases with increasing test

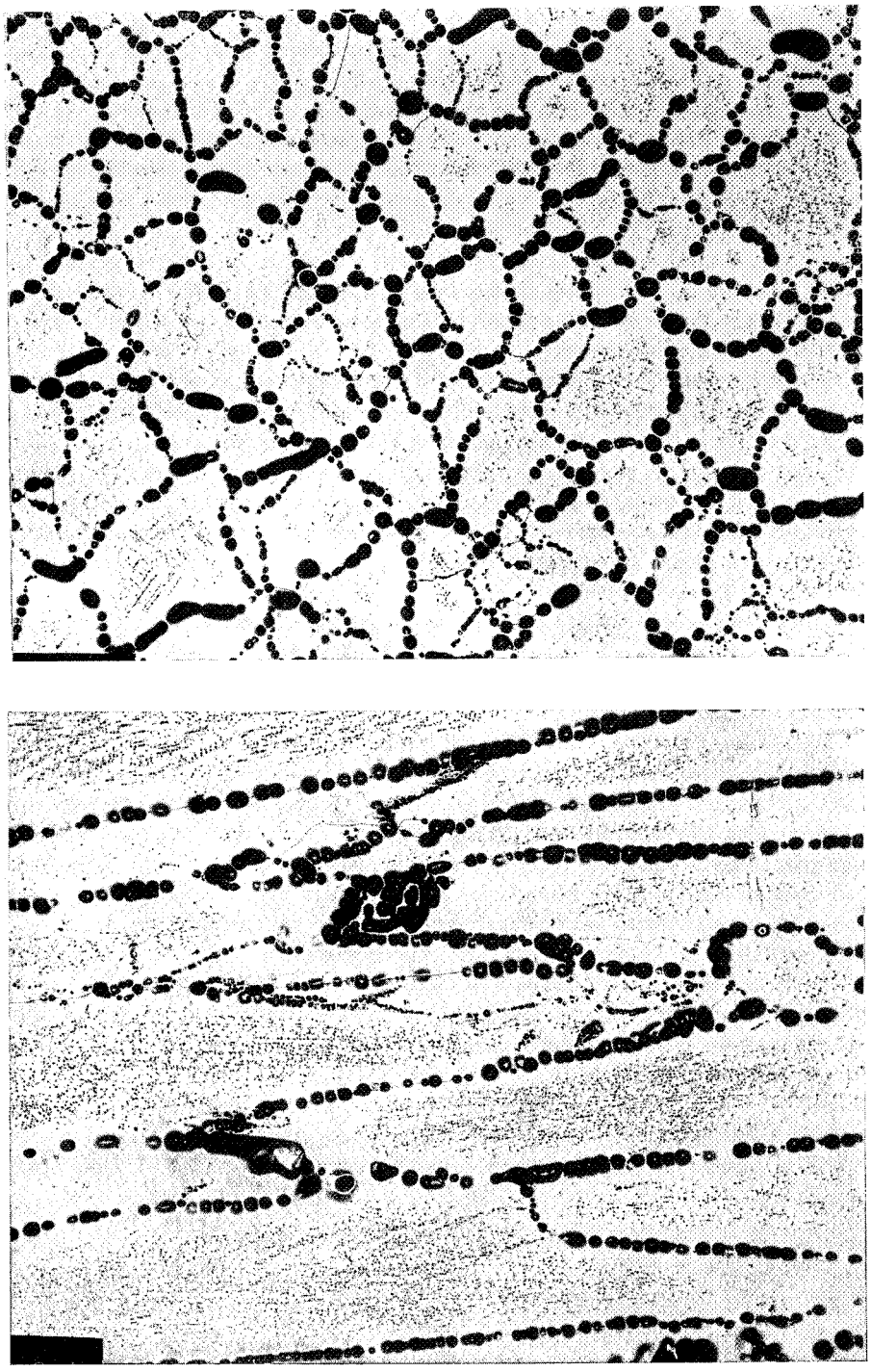


Fig. 8. Pyrolytic tungsten tested at 1930°C. Etched 1 min in 30 cm^3 lactic acid, 10 cm^3 HNO_3, and 10 cm^3 HF (× 100). (a) Section parallel to deposition plane. (b) Section normal to deposition plane.

temperatures, the reduction-in-area values remain relatively constant. It is thought that the tendency to increasing ductility with increasing temperature in some way offsets the effect of increasing void volume.

Elongation data are presented in Fig. 7. Although pyrolytic tungsten has lower values than the PM material, the shapes of the two curves are similar. PF material shows an increase in elongation throughout the temperature range studied. The slight increase in elongation in the PM material above approximately 2200°C was attributed to stress-induced grain growth[3]. Although little or no grain size change is observed in the pyrolytic material during testing, it exhibits a slight increase in elongation above 2200°C, followed by a decrease above 2480°C. Reasons for lower elongation values above approximately 2500°C for pyrolytic tungsten and above approximately 2750°C for PM tungsten are unknown.

Although insufficient pyrolytic material was available for extensive testing, a limited study of the formation of voids resulting from high-temperature heating was conducted. Void formation has been reported in PM tungsten wire rapidly heated above 2850°C in vacuum[4]. A vacuum pretreatment at lower temperatures eliminated the formation of voids on subsequent heating to higher temperature. Similarly, heating a pyrolytic tungsten specimen to 2200°C produced a microstructure containing no voids and with grains of approximately the same diameter (0.14 mm) as resulted from the 2845°C heat treatment.

One specimen, with a slightly modified gage diameter, was given a 2200°C anneal for 20 min and tested at 1930°C. Data are plotted with a filled triangle symbol (▲) in Figs. 5, 6, and 7 to compare with pyrolytic tungsten annealed at 2845°C. Tensile strength (Fig. 5) is seen to be slightly higher, presumably because of the absence of voids in the specimen prior to testing. Fracture occurred in an intercrystalline mode, and voids were observed in the microstructure after testing. Reduction-in-area and elongation values (Figs. 6 and 7) are found to be unchanged or even slightly lower. This suggests, on the basis of one test, that voids present after the high-temperature anneal have a relatively minor effect on ductility compared with voids formed during testing. As previously noted, the presence of grain boundaries perpendicular to the tensile axis appears to enhance the sensitivity to void formation and intercrystalline fracture.

In PM materials, detrimental substitutional impurities such as iron have been singled out as being a possible cause of void nucleation and growth[5]. This pyrolytic material, even with low substitutional impurity levels, still shows void formation and growth. However, grain boundary orientation, and possibly entrapped fluorine, may have some overriding effect.

It should be emphasized that these data are for one limited lot of pyrolytic tungsten. Tungsten deposited at other rates and temperatures may have different properties as varying conditions may be expected to affect grain size and, to a lesser extent, impurity level.

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